



Friction and Wear Properties of Selected Solid Lubricating Films

Part 3: Magnetron-Sputtered and Plasma-Assisted, Chemical-Vapor-Deposited Diamondlike Carbon Films

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SUMMARY

To evaluate commercially developed dry solid film lubricants for aerospace bearing applications, an investigation was conducted to examine the friction and wear behavior of magnetron-sputtered diamondlike carbon (MS DLC) and plasma-assisted, chemical-vapor-deposited diamondlike carbon (PACVD DLC) films in sliding contact with 6-mm-diameter American Iron and Steel Institute (AISI) 440C stainless steel balls. Unidirectional sliding friction experiments were conducted with a load of 5.9 N (600 g), a mean Hertzian contact pressure of 0.79 GPa (maximum Hertzian contact pressure of 1.2 GPa), and a sliding velocity of 0.2 m/s. The experiments were conducted at room temperature in three environments: ultrahigh vacuum (vacuum pressure, 7×10^{-7} Pa), humid air (relative humidity, ~20 percent), and dry nitrogen (relative humidity, <1 percent). The resultant films were characterized by scanning electron microscopy, energy-dispersive x-ray spectroscopy, and surface profilometry.

Marked differences in the friction and wear of the DLC films investigated herein resulted from the environmental conditions. The main criteria for judging the performance of the DLC films were coefficient of friction and wear rate, which had to be less than 0.3 and on the order of 10^{-6} mm³/N·m or less, respectively. MS DLC films and PACVD DLC films met the criteria in humid air and dry nitrogen but failed in ultrahigh vacuum, where the coefficients of friction were greater than the criterion, 0.3. In sliding contact with 440C stainless steel balls in all three environments the PACVD DLC films exhibited better tribological performance (i.e., lower friction and wear) than the MS DLC films. All sliding involved adhesive transfer of wear materials: transfer of DLC wear debris to the counterpart 440C stainless steel and transfer of 440C stainless steel wear debris to the counterpart DLC film.

INTRODUCTION

Diamondlike carbon (DLC) can be divided into two closely related categories known as amorphous, nonhydrogenated DLC (a-DLC or a-C) and amorphous, hydrogenated DLC (H-DLC or a-C:H) (ref. 1). H-DLC contains a variable and appreciable amount of hydrogen. DLC can be considered as a metastable carbon produced as a thin film with a broad range of structures (primarily amorphous with variable sp²/sp³ bonding ratio) and compositions (variable hydrogen concentration). A DLC's properties can vary considerably as its structure and composition vary (refs. 2 to 5). Although it is a complex engineering job, it is often possible to control and tailor the properties of a DLC to fit a specific application and thus ensure its success as a tribological product. However, such control demands a fundamental understanding of the tribological properties of DLC films. The absence of this understanding can act as a brake in applying DLC to a new product and in developing the product.

Tribological applications of DLC coatings and films are already well established in a number of fast-growing markets, such as magnetic recording media, high-density magnetic recording disks and sliders (heads), process equipment (e.g., copy machines and digital video camcorders), abrasion-resistant optical products, medical devices, implant components (including hip joints and knee implants), packaging materials, electronic devices, plastic molds, gear pumps, stamping devices, forming dies, blades (e.g., razor blades and scalpel knives), engine parts (e.g.,

gudgeon pins), washers (e.g., grease-free ceramic faucet valve seats), seals, valves, gears, bearings, bushings, tools, and wear parts (refs. 6 to 9). The cost is generally similar to that of carbide or nitride films deposited by CVD or physical vapor deposition (PVD) techniques. The surface smoothness, high hardness, low coefficient of friction, low wear rate, and chemical inertness of DLC coatings and films, along with little restriction of geometry and size, make them well suited as solid lubricants for applications involving wear and friction.

In parts 1 and 2 of the investigation (refs. 10 and 11), four types of selected solid lubricating film were examined in ultrahigh vacuum, in humid air at a relative humidity of approximately 20 percent, and in dry nitrogen at a relative humidity of less than 1 percent. The four types were bonded molybdenum disulfide (MoS_2) films, magnetron-sputtered MoS_2 films, ion-plated silver films, and ion-plated lead films.

The present investigation (part 3) was conducted to examine the friction and wear properties of magnetron-sputtered diamondlike carbon (MS DLC) and plasma-assisted, chemical-vapor-deposited diamondlike carbon (PACVD DLC) films in the same manner as in the parts 1 and 2 investigations. Both MS DLC and PACVD DLC films can be considered as a-DLC (amorphous, nonhydrogenated DLC). Magnetron sputtering and plasma-assisted CVD permit close control of film deposition and thickness, can provide good adhesion to the substrate, and can produce multilayer coatings. Unidirectional pin-on-disk sliding friction experiments were conducted with 440C stainless steel balls in sliding contact with the solid lubricating films at room temperature in ultrahigh vacuum (7×10^{-7} Pa), in humid air (relative humidity, ~20 percent), and in dry nitrogen (relative humidity, <1 percent). The resultant solid lubricating films and their wear surfaces were characterized by scanning electron microscopy (SEM), energy-dispersive x-ray spectroscopy (EDX), and surface profilometry. SEM and EDX were used to determine the morphology and elemental composition of wear surfaces, wear debris, and wear of the balls. The sampling depth of EDX for elemental information ranged between 0.5 and 1 μm in this investigation. Surface profilometry was used to determine the surface morphology, roughness, and wear of the coatings.

SELECTED MATERIALS

Three specimens of each film type, MS DLC and PACVD DLC, produced on 440C stainless steel disk substrates were used in this investigation (table I). The MS DLC films had a multilayer structure and were prepared using two chromium targets, 6 tungsten carbide (WC) targets, and methane (CH_4) gas. Each multilayer film comprised WC layers (20 to 50 nm thick) alternating with carbon layers (20 to 50 nm thick). The Vickers hardness number was approximately 1000. The 2- to 3- μm -thick MS DLC films were relatively smooth, and their centerline-average roughness R_a , measured using a cutoff of 1 mm, was 43 nm with a standard deviation of 5.1 nm.

The PACVD DLC films were prepared using radiofrequency plasma and consisted of two layers. Each film comprised an approximately 2- μm -thick DLC layer on an approximately 2- μm -thick silicon-DLC underlayer. The top DLC layer was deposited using CH_4 gas at a total pressure of 8 Pa with a power of 1800 to 2000 W at -750 to -850 V for 120 min. The silicon-containing DLC underlayer was deposited using a mixture of CH_4 and $\text{C}_4\text{H}_{12}\text{Si}$ (tetramethylsilane) gases. The ratio of the concentrations of CH_4 and $\text{C}_4\text{H}_{12}\text{Si}$ used was 90:18 (std cm^3/min) at a total pressure of 10 Pa with a power of 1800 to 2000 W at -850 to -880 V for 60 min. The Vickers hardness number was 1600 to 1800. The 3- to 5- μm -thick PACVD DLC films were also relatively smooth and their R_a , measured using a cutoff of 1 mm, was 29 nm with a standard deviation of 3.2 nm. The 6-mm-diameter 440C stainless steel balls (grade number, 10) used were smooth having an R_a of 0.025 μm with a standard deviation of 0.02 μm or less.

EXPERIMENT

The pin-on-disk tribometer used in the investigation was mounted in a vacuum chamber (refs. 10 and 11). Unidirectional pin-on-disk sliding friction experiments were conducted at room temperature in ultrahigh vacuum (7×10^{-7} Pa), in humid air (relative humidity, ~20 percent), and in dry nitrogen (relative humidity, <1 percent). All experiments were conducted with 6-mm-diameter 440C stainless steel balls in sliding contact with the DLC films deposited on 440C stainless steel substrate disks. All experiments were conducted with a load of 5.9 N (600 g) at the sliding velocity of 0.2 m/s. The mean Hertzian contact pressure of the 440C stainless steel substrates in contact with the 440C stainless steel balls was approximately 0.79 GPa (maximum Hertzian contact pressure, 1.2 GPa). The pin-on-disk tribometer can measure friction in vacuum, in humid air, and in dry nitrogen during sliding. The friction force was continuously monitored during the sliding friction experiments.

The sliding wear life (film wear life or film endurance life) for the coatings in this investigation was determined to be the number of passes at which the coefficient of friction rose to 0.3 in a given environment. Wear was quantified by measuring the wear scars and wear tracks on the specimens after the wear experiments. Film wear volumes were obtained by averaging the cross-sectional areas, determined from stylus tracings, measured across the wear tracks at a minimum of four locations in each wear track. Then, the average cross-sectional area of the wear track was multiplied by the wear track length. The wear rate, known as the dimensional wear coefficient, is defined as the volume of material removed at a unit load and in a unit sliding distance expressed as cubic millimeters per newton-meter.

RESULTS AND DISCUSSION

Friction Behavior

Figures 1 to 3 present typical friction traces obtained in ultrahigh vacuum, in humid air, and in dry nitrogen, respectively, for the MS DLC and PACVD DLC films in sliding contact with 440C stainless steel balls as a function of the number of passes. All the friction traces for the DLC films obtained in the three environments fluctuated. In ultrahigh vacuum (fig. 1) the coefficient of friction for both the MS DLC and PACVD DLC films rose to 0.3 in a few passes; the steady-state values were approximately 0.7 for the MS DLC films and 0.54 for the PACVD DLC films. In humid air (fig. 2) the coefficients of friction for both the MS DLC and PACVD DLC films decreased to approximately 0.1; the steady-state value for the PACVD DLC films was generally lower than that for the MS DLC films. In dry nitrogen (fig. 3) the coefficient of friction for the MS DLC films increased to 0.3 at approximately 24 000 passes, and the steady-state coefficient of friction for the PACVD DLC films decreased to 0.05 at 300 000 passes.

Comparing the data taken in the different environments (figs. 1 to 3) shows that the coefficients of friction for both the MS DLC and PACVD DLC films were much higher in ultrahigh vacuum than in humid air and in dry nitrogen. The coefficients of friction of the PACVD DLC films were generally lower than those of the MS DLC films in all three environments.

Wear Behavior

Figures 4 to 6 present SEM photomicrographs of wear tracks on the MS DLC and PACVD DLC films deposited on 440C stainless steel disks and the wear scars on the 440C stainless steel balls in ultrahigh vacuum, in humid air, and in dry nitrogen, respectively. The SEM observations were made at 1000 passes in ultrahigh vacuum, at 300 000 passes in humid air, and either at the end of film wear life or at 300 000 passes in dry nitrogen. In ultrahigh vacuum (fig. 4) the sliding action roughened the entire wear tracks of the MS DLC films at 1000 passes but locally produced micro-pits in the wear tracks of the PACVD DLC films. With both films types in ultrahigh vacuum, wear debris particles and agglomerated wear debris were generated during sliding. In humid air the sliding action generated smooth wear surfaces on the MS DLC films and deposited a large amount of agglomerated, pasty wear debris on the wear scars of the 440C stainless steel balls (fig. 5(a)). In humid air the sliding action generated a smooth wear surface on the PACVD DLC films with relatively large wear debris particles (fig. 5(b)). In dry nitrogen the sliding action generated a smooth wear surface for both film types and produced fine wear debris particles with the MS DLC films (fig. 6(a)) and relatively large wear debris particles with the PACVD DLC films (fig. 6(b)).

The wear scars on the 440C stainless steel balls were generally smooth, regardless of the environment. Thin, smeared wear patches and particles of the DLC films generally covered the smooth wear scars. Smeared tongues of thin, layered, agglomerated wear debris were also present. Most of the loose and smeared wear debris accumulated outside the wear scars.

Wear (Endurance) Life

As in parts 1 and 2 of the investigation (refs. 10 and 11) the sliding wear (endurance) life of the solid lubricating films deposited on 440C stainless steel disks was determined to be the number of passes at which the coefficient of

friction rose to 0.3. The sliding wear lives of the DLC films examined in this investigation (table II) varied with the environment. When judged by the coefficient of friction, the wear lives of both film types were extremely short in ultrahigh vacuum. The MS DLC films had much longer wear lives in humid air than in dry nitrogen and in ultrahigh vacuum. The PACVD DLC films had much longer wear lives in humid air and in dry nitrogen than in ultrahigh vacuum.

Comparison of Steady-State Coefficients of Friction and Wear Rates

Table II also presents the steady-state coefficients of friction and the film and ball wear rates after sliding contact in all three environments. The data presented in the table reveal the marked differences in coefficient of friction resulting from the environmental conditions. Both the MS DLC and PACVD DLC films had high coefficients of friction, high film wear rates, and high ball wear rates in ultrahigh vacuum but relatively low coefficients of friction, low film wear rates, and low ball wear rates in humid air and in dry nitrogen. Both film types met the main criteria for judging the tribological performance of films (coefficient of friction less than 0.3 and wear rate on the order of 10^{-6} mm³/N·m or less) in humid air and in dry nitrogen. In sliding contact with a 440C stainless steel ball the PACVD DLC films exhibited better tribological performance (i.e., lower friction and wear) than did the MS DLC films in all three environments.

Sliding Wear, Wear Debris, and Transferred Wear Fragments

Examining the morphology and composition of the worn surfaces of MS DLC and PACVD DLC films in sliding contact with 440C stainless steel balls by SEM and EDX provided detailed information about plastic deformation of the DLC films, wear debris, and transferred wear fragments produced during sliding (figs. 7 to 12). All sliding involved generation of fine wear debris particles and agglomerated wear debris and transfer of the worn materials.

Ultra-high-vacuum environment.—Figure 7(a) presents a typical wear track on an MS DLC film over which a 440C stainless steel ball has passed in ultrahigh vacuum leaving a roughened worn DLC film surface and a small amount of transferred steel wear fragments. The wear scar on the counterpart 440C stainless steel ball (fig. 7(b)) contained fine steel wear debris particles and a small amount of transferred DLC wear fragments. The wear mechanism for an MS DLC film in sliding contact with a 440C stainless steel ball in ultrahigh vacuum is that of small DLC fragments chipping off the surface.

Figure 8(a) presents a typical wear track on a PACVD DLC film over which a 440C stainless steel ball has passed in ultrahigh vacuum leaving smeared, agglomerated DLC wear debris and a small amount of transferred steel wear fragments. The wear scar on the counterpart 440C stainless steel ball (fig. 8(b)) contained fine steel wear debris particles and large smeared, agglomerated wear debris patches containing transferred DLC wear fragments. The wear mechanism for a PACVD DLC film in sliding contact with a 440C stainless steel ball in ultrahigh vacuum was adhesion, and plastic deformation played a role in the burnished appearance of the agglomerated wear debris.

Humid-air environment.—Figure 9(a) presents a typical wear track on an MS DLC film over which a 440C stainless steel ball has passed in humid air leaving a small amount of transferred steel wear fragments. The fine asperities of the MS DLC film were flattened and elongated in the sliding direction by plastic deformation, revealing a smooth, burnished appearance. The entire wear scar on the counterpart 440C stainless steel ball (fig. 9(b)) contained thick transferred layers (or sheets) of MS DLC. Plate-like DLC wear debris particles were found at the edges of the wear scar. Severe plastic deformation and shearing occurred in the DLC film during sliding.

Figure 10(a) presents a typical wear track on a PACVD DLC film over which a 440C stainless steel ball has passed in humid air leaving a small amount of transferred steel wear fragments. The fine asperities of the PACVD DLC film were flattened and elongated in the sliding direction by plastic deformation, revealing a smooth, burnished appearance. The smooth wear scar on the counterpart 440C stainless steel ball (fig. 10(b)) contained an extremely small amount of transferred PACVD DLC wear debris.

Dry-nitrogen environment.—Figure 11(a) presents a typical wear track on an MS DLC film over which a 440C stainless steel ball has passed in dry nitrogen. At 23 965 passes (end of life) there was an extremely small amount of transferred steel wear debris, and the fine asperities of the MS DLC film were flattened and elongated in the sliding direction by plastic deformation, revealing a smooth, burnished appearance. In addition to the small amount of steel

wear debris particles, smeared, agglomerated DLC wear debris was found on the MS DLC film. Plastic deformation occurred in the DLC film during sliding. The wear scar on the counterpart 440C stainless steel ball (fig. 11(b)) contained transferred DLC wear debris particles and patches.

Figure 12(a) presents a typical wear track on a PACVD DLC film over which a 440C stainless steel ball has passed in dry nitrogen. At 300 000 passes DLC wear debris, micro-pits, and an extremely small amount of transferred steel wear debris were observed. The wear scar on the counterpart 440C stainless steel ball (fig. 12(b)) contained fine grooves in the sliding direction, steel wear debris, and transferred DLC wear debris.

CONCLUSIONS

To evaluate recently developed diamondlike carbon (DLC) film lubricants for aerospace bearing applications, unidirectional sliding friction experiments were conducted with DLC films in sliding contact with AISI 440C stainless steel balls in ultrahigh vacuum, in humid air, and in dry nitrogen. The main criteria for judging the performance of the DLC films were coefficient of friction and wear rate, which had to be less than 0.3 and on the order of 10^{-6} mm³/N·m or less, respectively. The following conclusions were drawn:

1. Magnetron-sputtered (MS) DLC films and plasma-assisted, chemical-vapor-deposited (PACVD) DLC films met the criteria in humid air and in dry nitrogen but failed in ultrahigh vacuum, where the coefficients of friction were greater than the criterion, 0.3.
2. In sliding contact with a 440C stainless steel ball the PACVD DLC films exhibited better tribological performance (i.e., lower friction and wear) in all three environments than the MS DLC films.
3. All sliding involved adhesive transfer of wear materials: transfer of DLC wear debris to the counterpart 440C stainless steel ball and transfer of 440C stainless steel wear debris to the counterpart DLC film.

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TABLE I.—CHARACTERISTICS OF SELECTED SOLID
LUBRICATING FILMS
[Film material, carbon; substrate material, 440C stainless steel.]

Film type	Film thickness, μm	Surface roughness of films, R_a , nm	
		Mean	Standard deviation
Magnetron-sputtered diamondlike carbon (MS DLC)	2–3	43	5.1
Plasma-assisted, chemical-vapor-deposited diamondlike carbon (PACVD DLC)	3–5	29	3.2

TABLE II.—STEADY-STATE COEFFICIENT OF FRICTION, WEAR LIFE,
AND WEAR RATES FOR DLC FILMS IN SLIDING CONTACT WITH
440C STAINLESS STEEL BALLS

Film	Environment	Steady-state coefficient of friction	Film wear (endurance) life ^a	Film wear rate, $\text{mm}^3/\text{N}\cdot\text{m}$	Ball wear rate, $\text{mm}^3/\text{N}\cdot\text{m}$
Magnetron-sputtered DLC	Vacuum	0.70	<10	5.7×10^{-5}	3.2×10^{-4}
	Air	0.12	$>3 \times 10^5$	1.7×10^{-7}	4.1×10^{-8}
	Nitrogen	0.12	23 965	4.2×10^{-7}	1.1×10^{-7}
Plasma-assisted CVD DLC	Vacuum	0.54	<10	1.1×10^{-5}	1.8×10^{-4}
	Air	0.07	$>3 \times 10^5$	1.0×10^{-7}	2.3×10^{-8}
	Nitrogen	0.06	$>3 \times 10^5$	1.1×10^{-8}	6.4×10^{-9}

^aFilm wear life is determined to be the number of passes at which the coefficient of friction rose to 0.3.

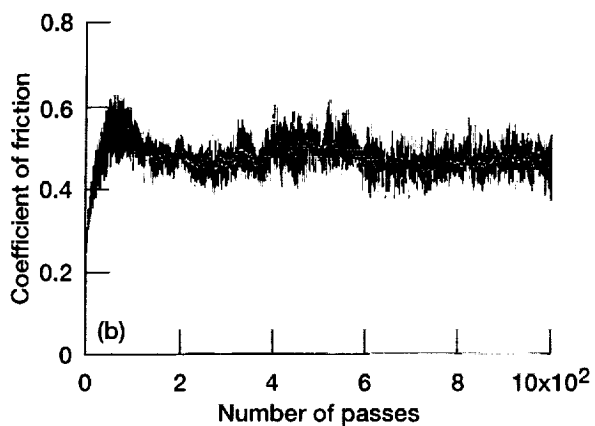
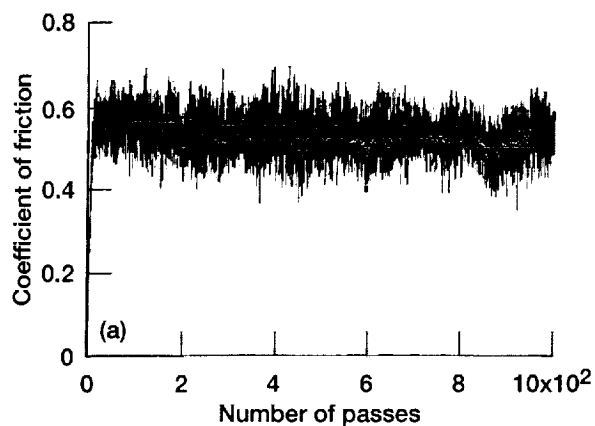


Figure 1.—Friction traces for (a) MS DLC film and (b) PACVD DLC film in sliding contact with 440C stainless steel balls in ultrahigh vacuum.

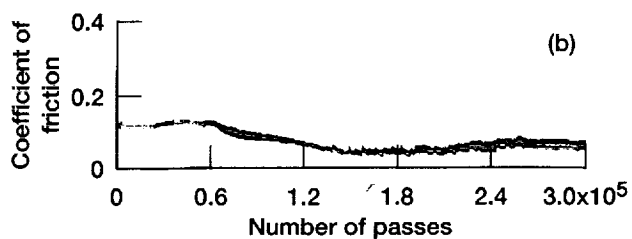
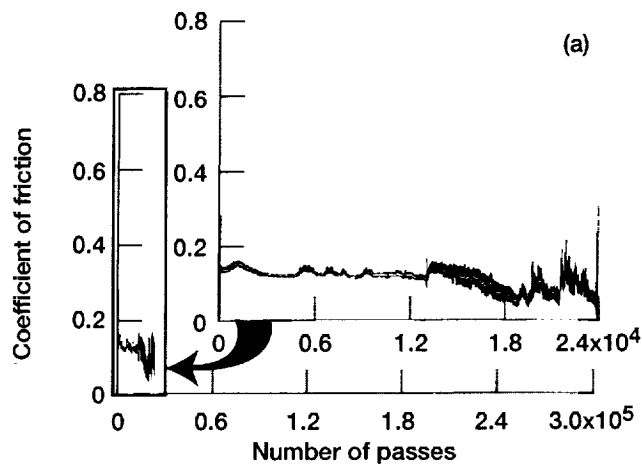


Figure 3.—Friction traces for (a) MS DLC film and (b) PACVD DLC film in sliding contact with 440C stainless steel balls in dry nitrogen.

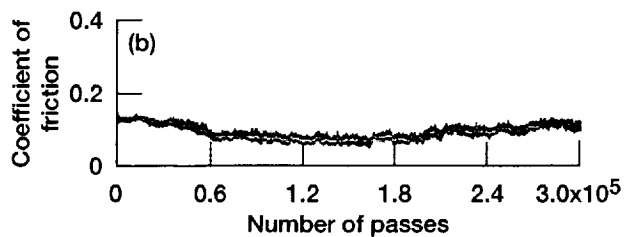
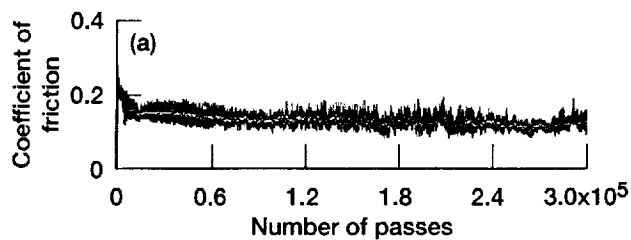


Figure 2.—Friction traces for (a) MS DLC film and (b) PACVD DLC film in sliding contact with 440C stainless steel balls in humid air.

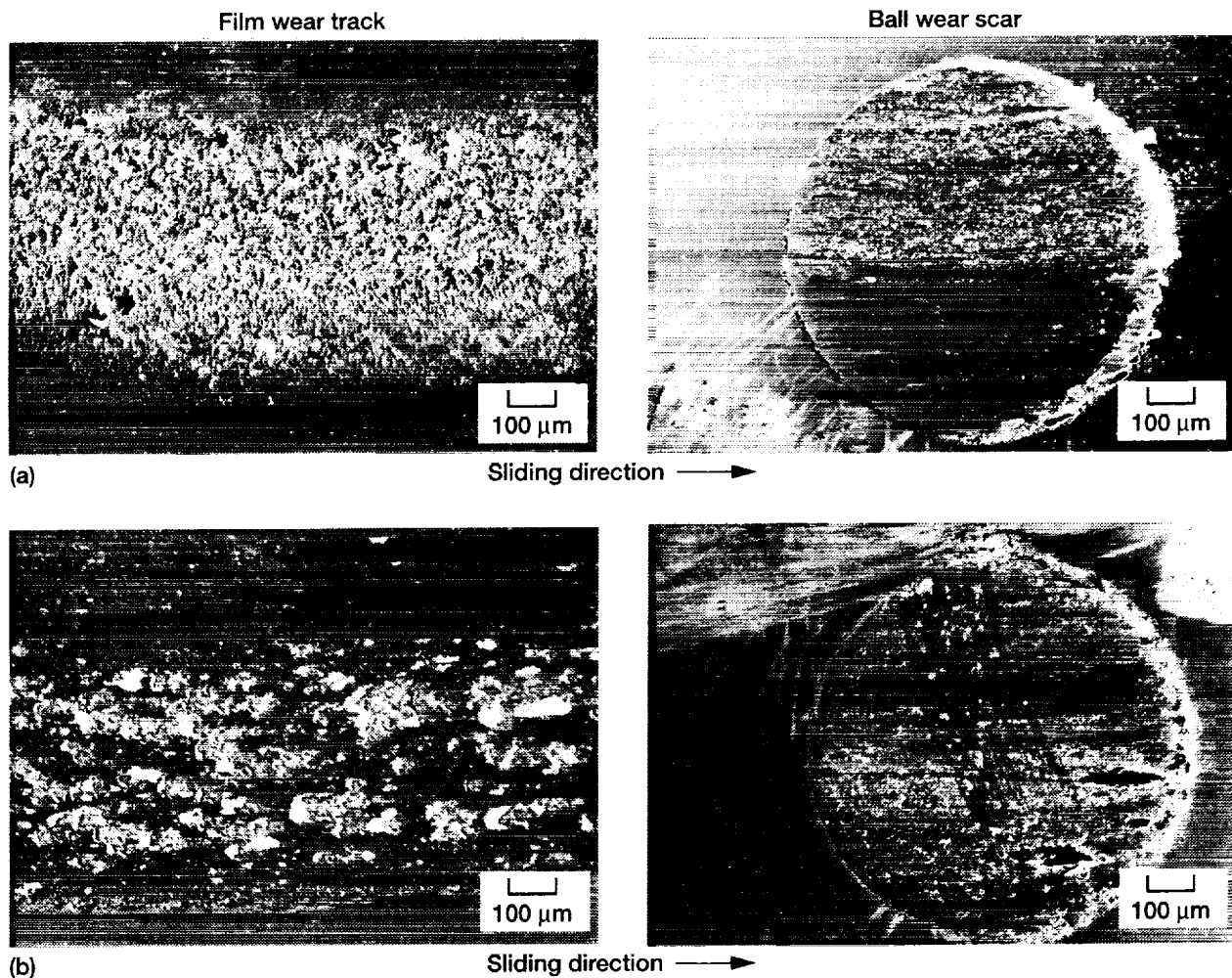


Figure 4.—Wear tracks and wear scars in ultrahigh vacuum at 1000 passes. (a) Materials pair of MS DLC film and 440C stainless steel ball. (b) Materials pair of PACVD DLC film and 440C stainless steel ball.

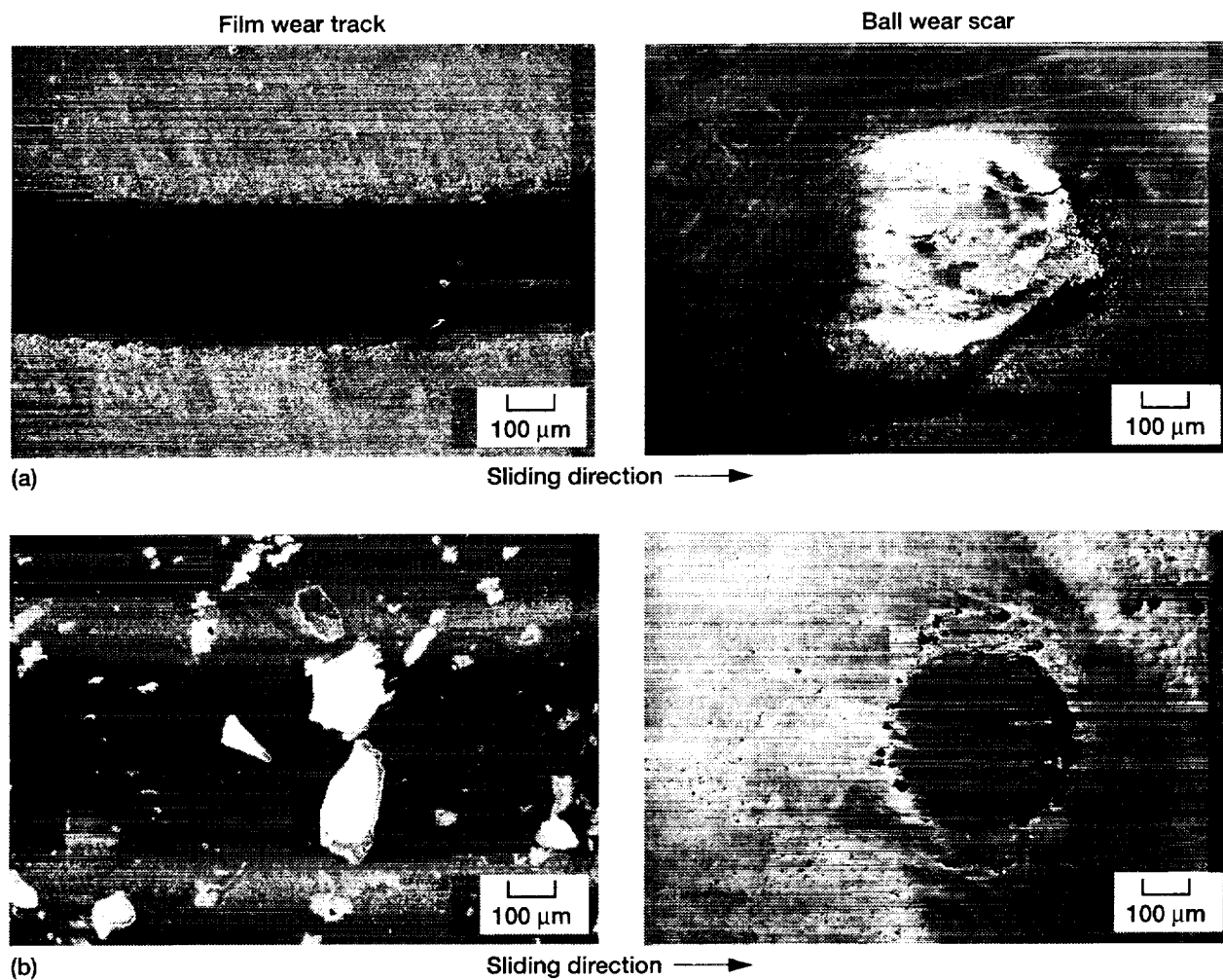


Figure 5.—Wear tracks and wear scars in humid air at 300 000 passes. (a) Materials pair of MS DLC film and 440C stainless steel ball. (b) Materials pair of PACVD DLC film and 440C stainless steel ball.

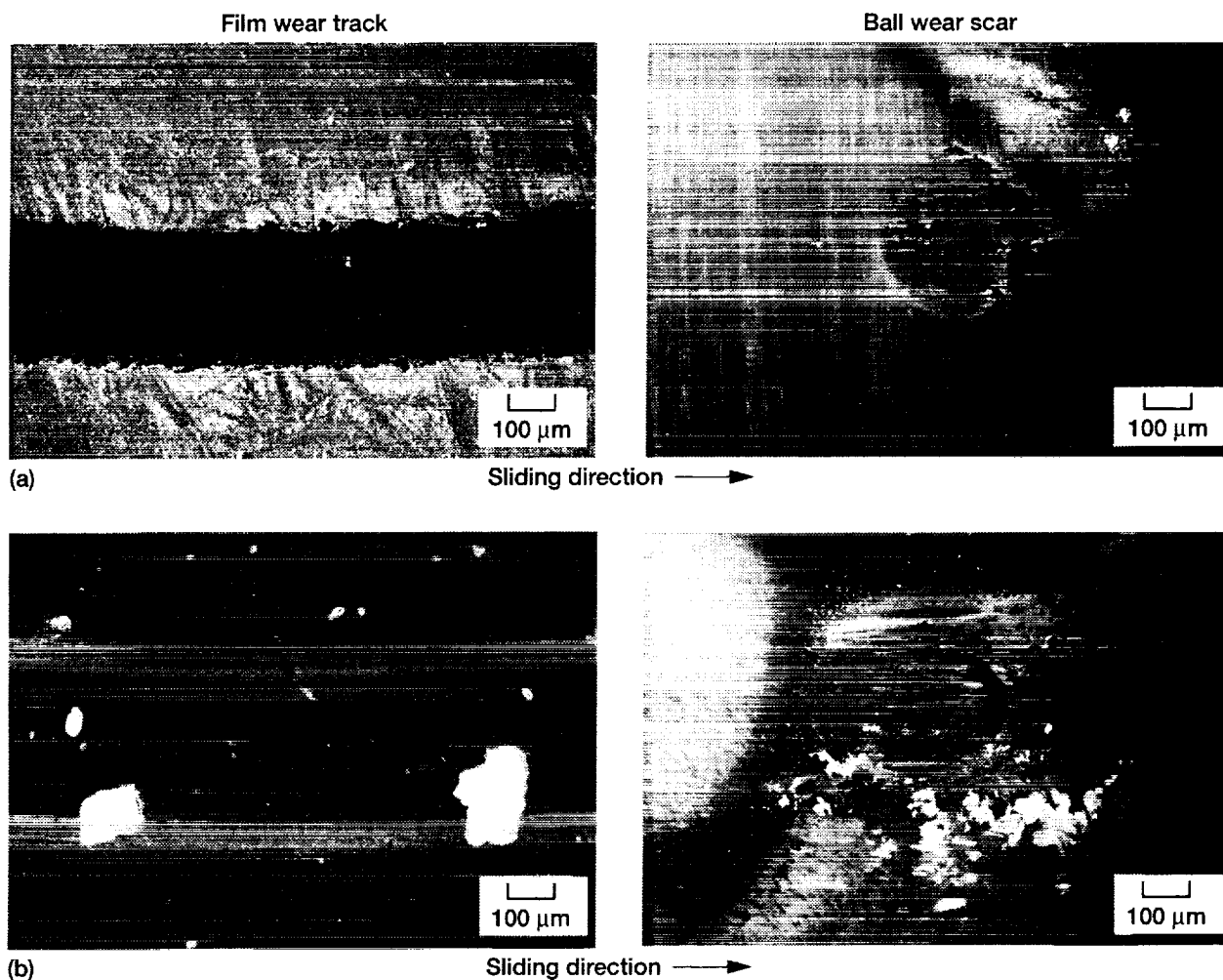
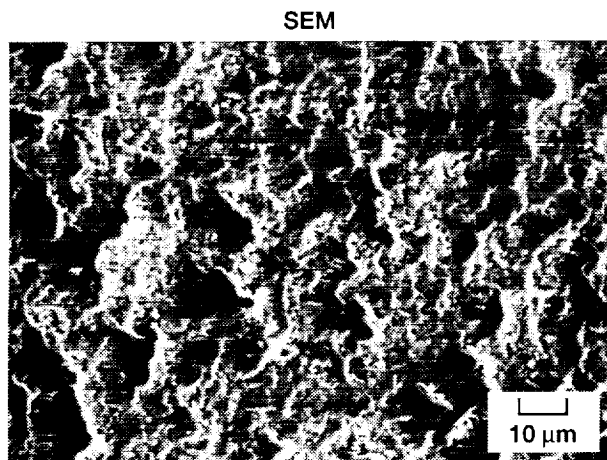
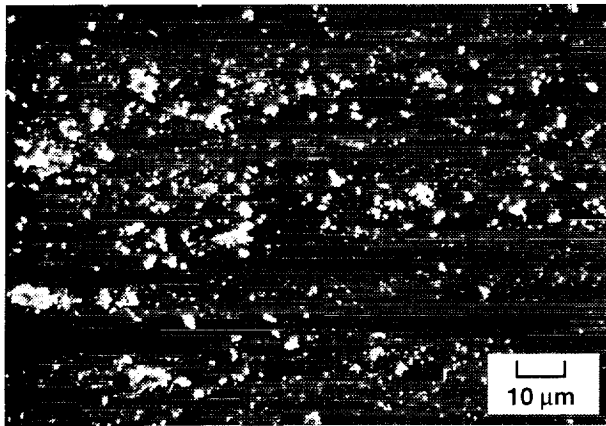
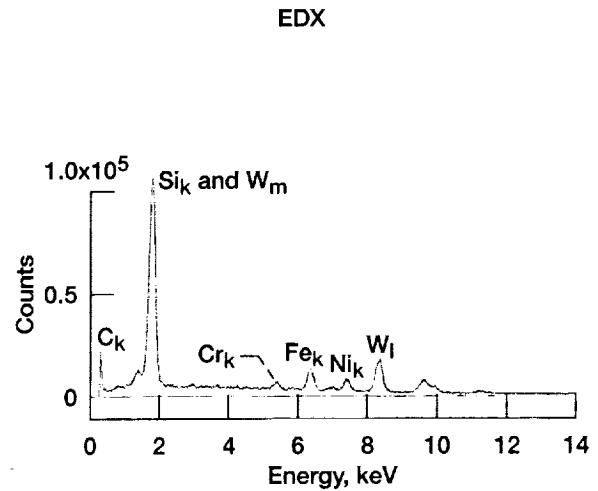


Figure 6.—Wear tracks and wear scars in dry nitrogen. (a) Materials pair of MS DLC film and 440C stainless steel ball at 23 965 passes. (b) Materials pair of PACVD DLC film and 440C stainless steel ball at 300 000 passes.



(a)



(b)

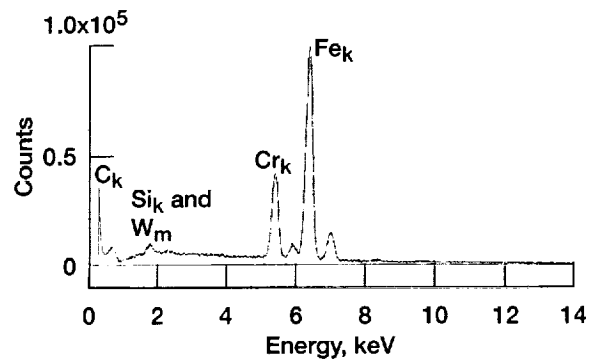


Figure 7.—Morphology and elemental composition by SEM and EDX (a) of wear track produced on MS DLC film and (b) of wear scar produced on 440C stainless steel ball at 1000 passes in ultrahigh vacuum.

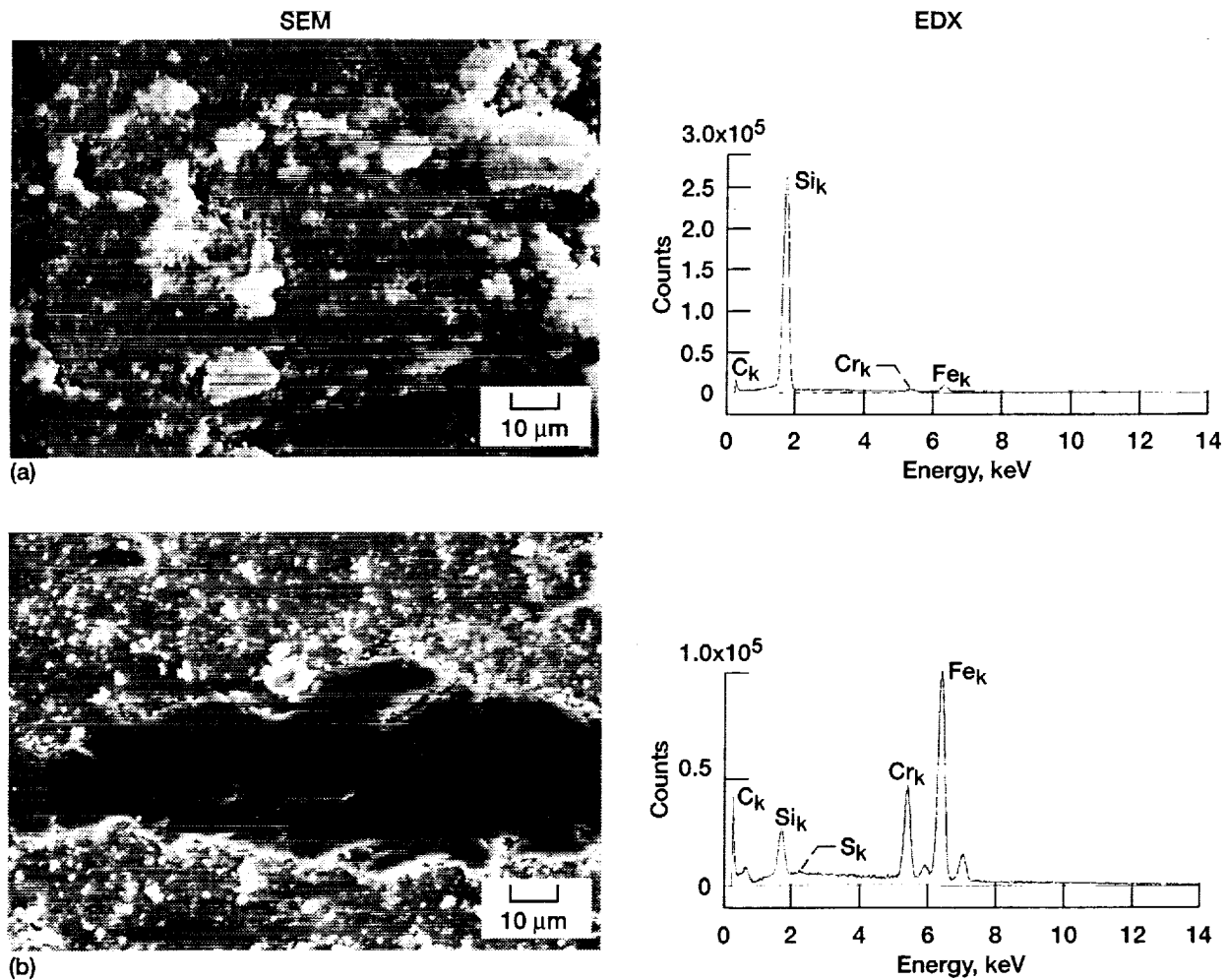


Figure 8.—Morphology and elemental composition by SEM and EDX (a) of wear track produced on PACVD DLC film and (b) of wear scar produced on 440C stainless steel ball at 1000 passes in ultrahigh vacuum.

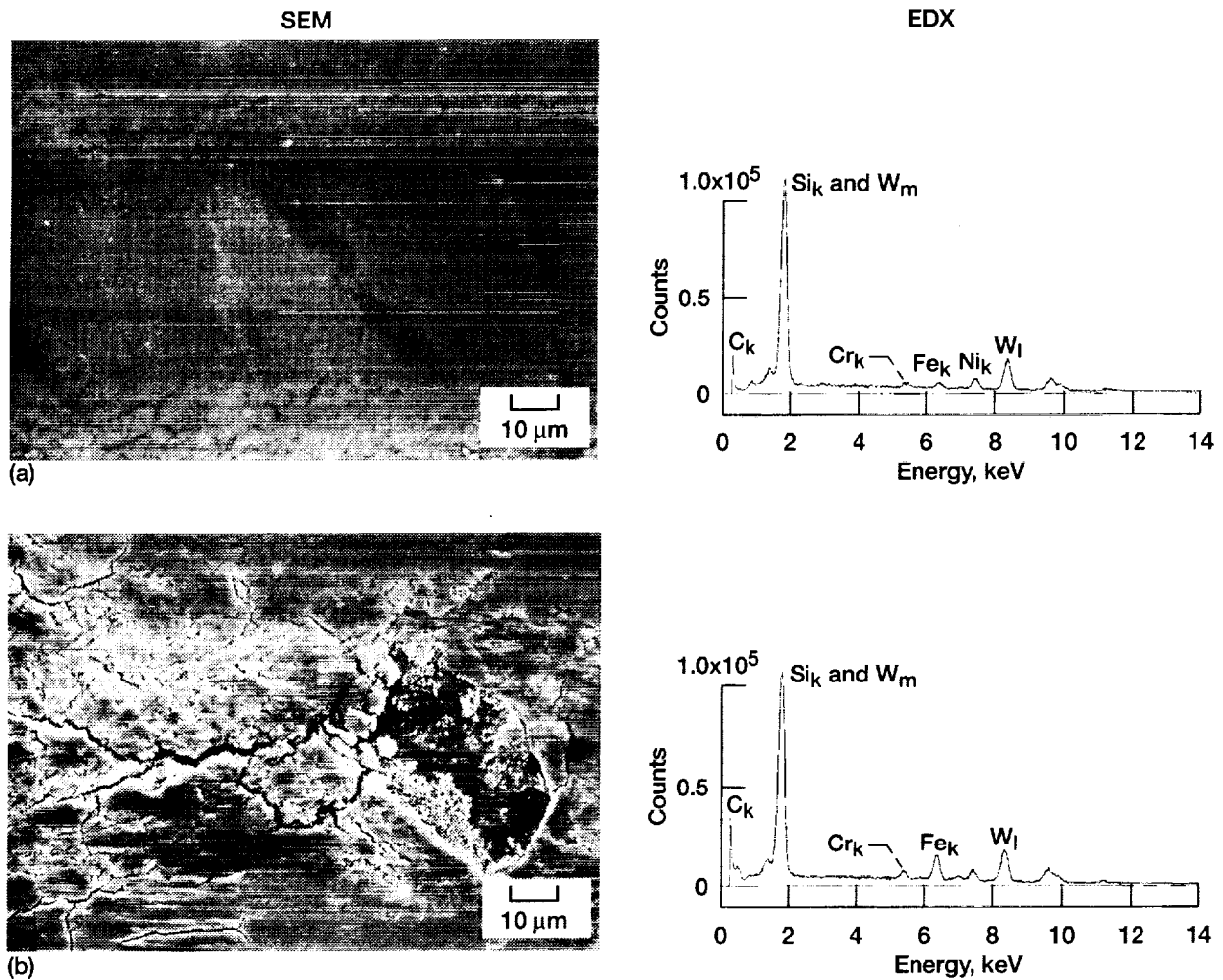


Figure 9.—Morphology and elemental composition by SEM and EDX (a) of wear track produced on MS DLC film and (b) of wear scar produced on 440C stainless steel ball at 300 000 passes in humid air.

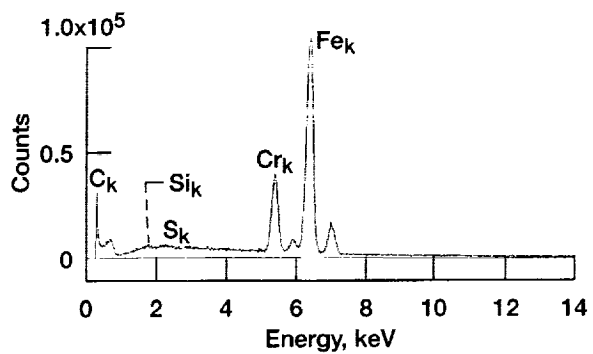
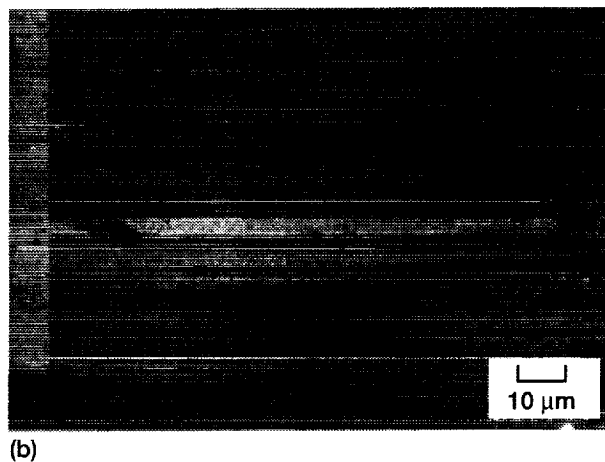
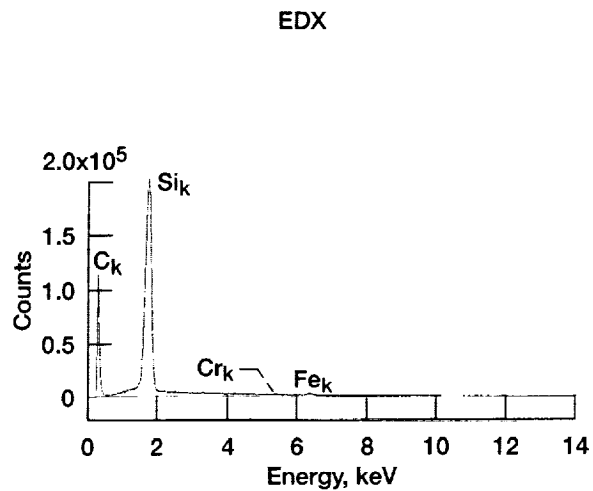
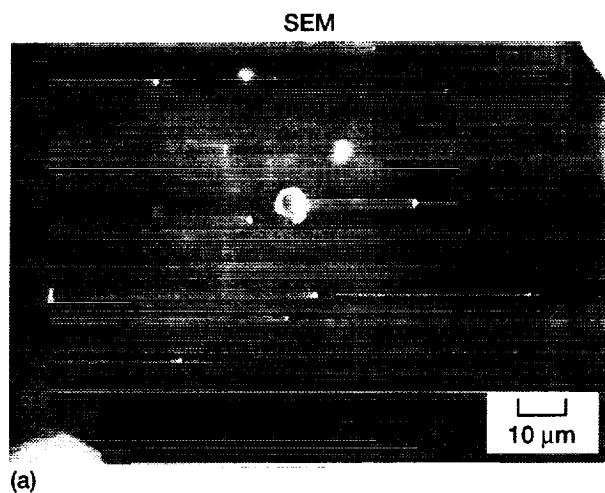


Figure 10.—Morphology and elemental composition by SEM and EDX (a) of wear track produced on PACVD DLC film and (b) of wear scar produced on 440C stainless steel ball at 300 000 passes in humid air.

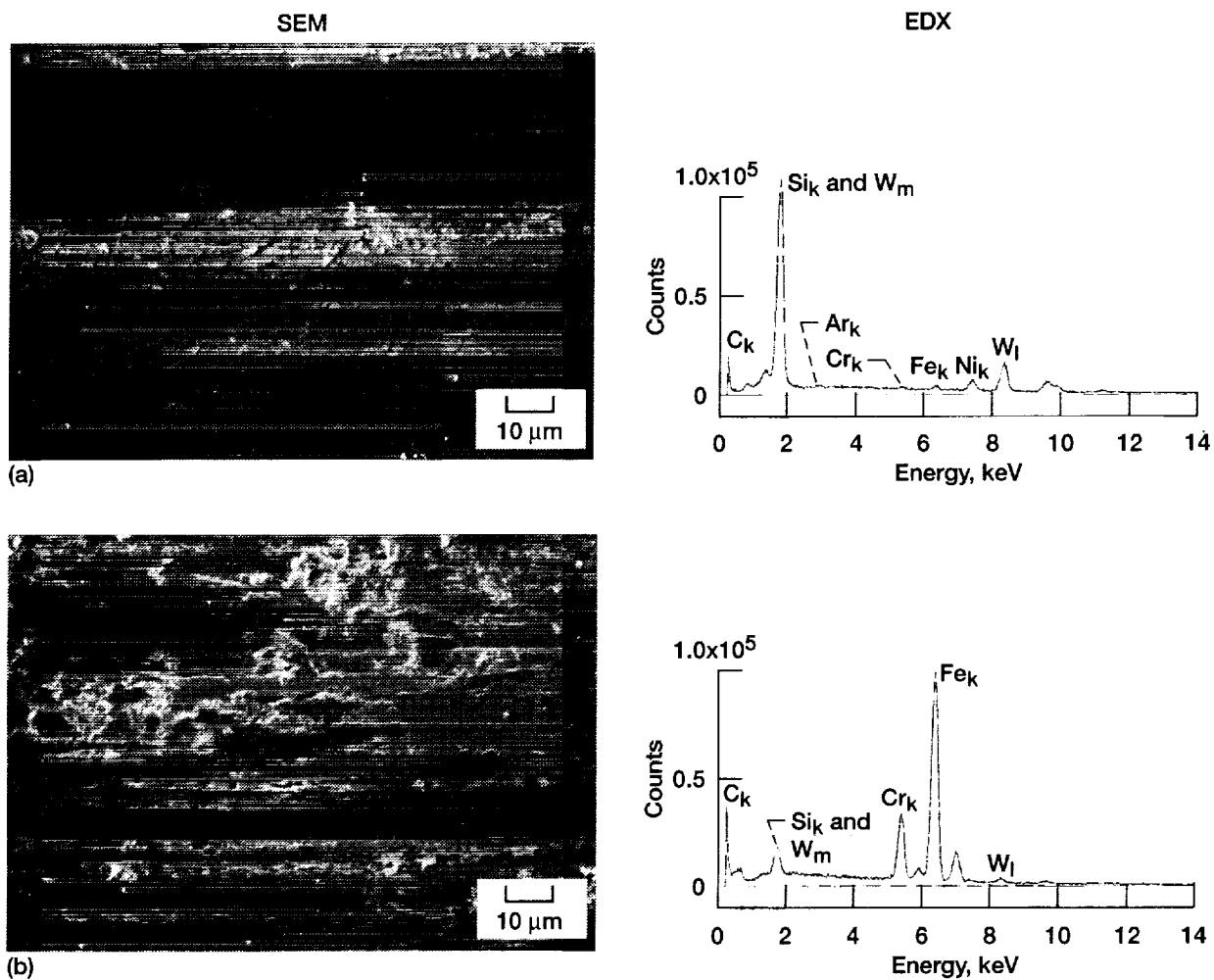
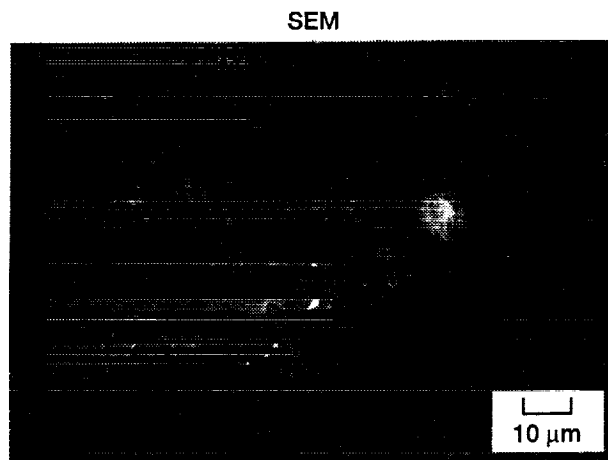
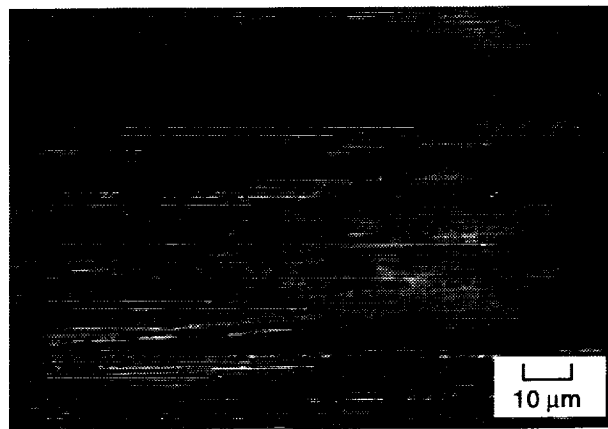
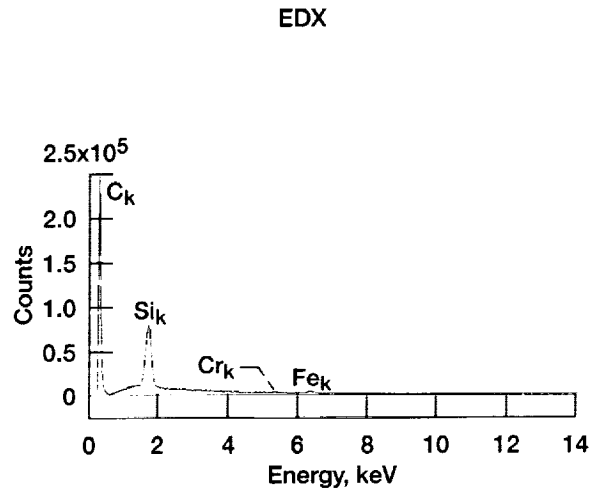


Figure 11.—Morphology and elemental composition by SEM and EDX (a) of wear track produced on MS DLC film and (b) of wear scar produced on 440C stainless steel ball at 23 965 passes in dry nitrogen



(a)



(b)

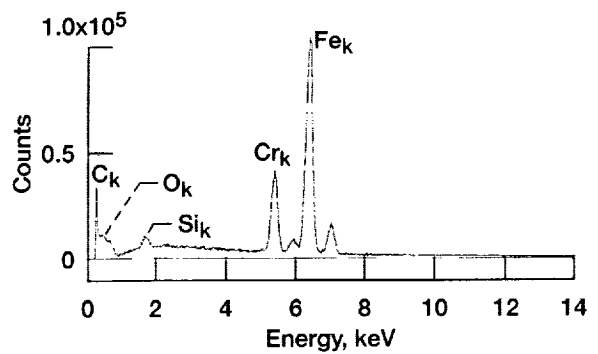


Figure 12.—Morphology and elemental composition by SEM and EDX (a) of wear track produced on PACVD DLC film and (b) of wear scar produced on 440C stainless steel ball at 300 000 passes in dry nitrogen.

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13. ABSTRACT (Maximum 200 words) To evaluate commercially developed dry solid film lubricants for aerospace bearing applications, an investigation was conducted to examine the friction and wear behavior of magnetron-sputtered diamondlike carbon (MS DLC) and plasma-assisted, chemical-vapor-deposited diamondlike carbon (PACVD DLC) films in sliding contact with 6-mm-diameter American Iron and Steel Institute (AISI) 440C stainless steel balls. Unidirectional sliding friction experiments were conducted with a load of 5.9 N (600 g), a mean Hertzian contact pressure of 0.79 GPa (maximum Hertzian contact pressure of 1.2 GPa), and a sliding velocity of 0.2 m/s. The experiments were conducted at room temperature in three environments: ultrahigh vacuum (vacuum pressure, 7×10^{-7} Pa), humid air (relative humidity, ~20 percent), and dry nitrogen (relative humidity, <1 percent). The resultant films were characterized by scanning electron microscopy, energy-dispersive x-ray spectroscopy, and surface profilometry. Marked differences in the friction and wear of the DLC films investigated herein resulted from the environmental conditions. The main criteria for judging the performance of the DLC films were coefficient of friction and wear rate, which had to be less than 0.3 and on the order of 10^{-6} mm ³ /N-m or less, respectively. MS DLC films and PACVD DLC films met the criteria in humid air and dry nitrogen but failed in ultrahigh vacuum, where the coefficients of friction were greater than the criterion, 0.3. In sliding contact with 440C stainless steel balls in all three environments the PACVD DLC films exhibited better tribological performance (i.e., lower friction and wear) than the MS DLC films. All sliding involved adhesive transfer of wear materials: transfer of DLC wear debris to the counterpart 440C stainless steel and transfer of 440C stainless steel wear debris to the counterpart DLC film.				
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